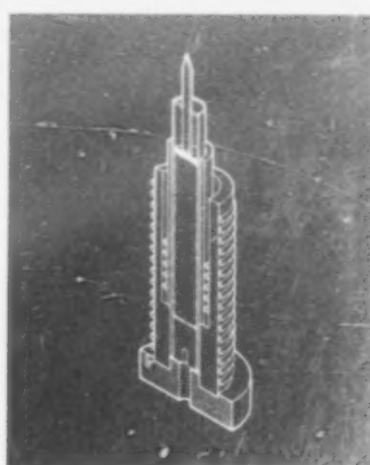
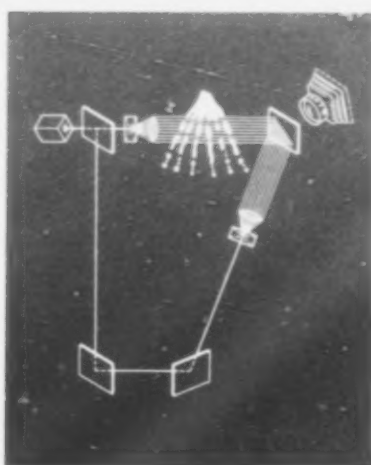
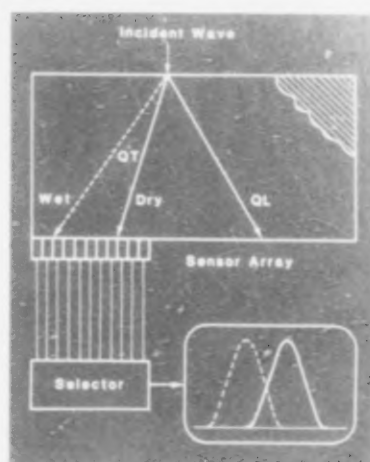
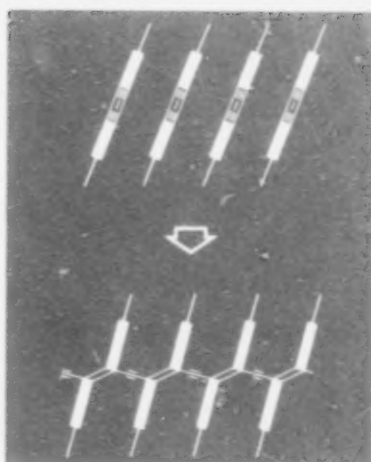
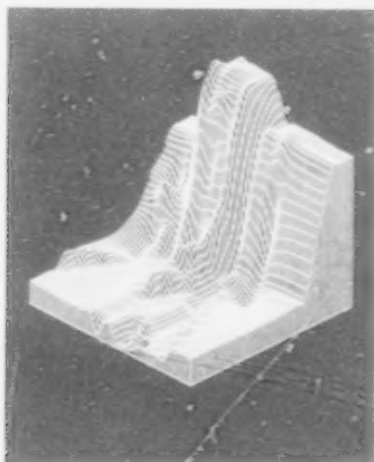




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Cover:

Schematic representation of the annual report covers for the operating divisions of the Materials Science and Engineering Laboratory and its Office of Nondestructive Evaluation. These annual reports describe in detail the technical activities of each of the Laboratory's major units and are available on request. Requests for the annual reports should be addressed to Lyle H. Schwartz, Director, Materials Science and Engineering Laboratory, B309 Materials Bldg., NIST, Gaithersburg, MD 20899.

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M **aterials Science and Engineering Laboratory**

Materials are so basic to human progress that their importance is hard to overemphasize. Every technological advance, from the first stone hammer to today's most complex integrated circuits, sprang from the mastery of specific materials. Accomplishing many of the nation's goals, such as the National Aerospace Plane, faster computers, or electrical energy from nuclear fusion, hinges on our ability to develop new materials that are far superior to those used today. Indeed, the technological and economic goals of many other countries are also tied to advances in materials, setting the stage for intense international competition. For example, more than half of Japan's 13 priority research and development projects involve materials science. In Europe, the Commission of European Communities has set up national and international cooperative programs that focus on advanced materials.

Research on advanced ceramics, metal alloys, polymers, composites, and the new high-temperature superconductors has already exposed some of the future technological possibilities. The ability to microengineer these materials—to create submicroscopic structures and combinations of elements unknown in nature—has resulted in properties and phenomena that until only recently were beyond the imagination. Now that our imaginations have been piqued and our appetites for innovation whetted, our challenge is to transform these materials from laboratory curiosities to viable products and manufacturing processes.

At the National Institute of Standards and Technology (formerly the National Bureau of Standards), the Materials Science and Engineering Laboratory (MSEL) is responding to this challenge in many ways, addressing scientific and measurement issues that are crucial to industry's success in exploiting the potential technological advantages of advanced materials. Consistent with the Institute's mission as the national measurement and engineering laboratory, the activities of MSEL's six units emphasize research leading to the development of standards, test methods, and reference data and materials. The guiding tenet for this work is a simple maxim: If a process or property cannot be measured, then it is not completely understood. Inherent in MSEL's mission is the need to develop scientific understanding of the underlying physical and chemical origins of the properties being evaluated.

Covering all classes of advanced materials, as well as major conventional materials, the Laboratory's research program investigates the relationship between the structure and properties of materials and then applies the resulting knowledge to issues related to the design, processing, and performance of materials. Our approach encompasses the full range of research and development activities, from basic studies to generic applications. The examples of MSEL research accomplishments described in this publication illustrate this R&D strategy.

Toward Automated Process Control

In recent years, the focus of the Laboratory's attention has increasingly turned toward improving the understanding of materials processing. Our ultimate goal is to help U.S. industry develop real-time, automated systems of process control, so-called intelligent manufacturing methods that improve product quality and increase production

efficiency. As study after study has pointed out, firms that emphasize product quality throughout the manufacturing process, rather than at the end of the line only, will strengthen their competitive position.

Intelligent processing of materials is itself the result of a process, the evolutionary development and application of scientific and engineering knowledge. Projects under way in the five MSEL divisions and those managed by the Office of Nondestructive Evaluation are gathering and evaluating much-needed data on structures and properties of materials, at increasingly fine levels of detail and under the increasingly extreme environmental conditions that characterize many high-performance applications. These data support the development of process models, which relate the specific properties of materials at each manufacturing step to the properties of the final product.

In tandem with data-gathering and modeling efforts, MSEL researchers are building and refining real-time sensors for in-process nondestructive measurements. They then develop strategies for coupling on-line measurements with process models. This work establishes the foundation for the final step—merging sensors and models with automation technology to create a truly integrated manufacturing system. Without closed-loop process control, manufacturing methods will be inefficient, often failing to achieve the carefully controlled microstructures that govern the performance and reliability of advanced materials.

As you will see, process control is the common thread that unifies the Laboratory's varied technical activities—from the development and refinement of theory to innovative methods for on-line nondestructive evaluation. Our work exemplifies the increasingly close link between basic scientific understanding and technological innovation.

Staff and Facilities

MSEL has a research staff of nearly 400 scientists, engineers, and technicians, supported by a budget of about \$46 million in 1989. Their efforts are enhanced by the broad range of scientific and engineering expertise and instrumentation available in other parts of NIST, at its facilities in Gaithersburg, Maryland, and Boulder, Colorado. Collaborations are common: In fact, the multidisciplinary makeup of NIST is an especially valuable asset in materials science and engineering, where answers to important research questions typically require contributions from physics, chemistry, the traditional materials-related disciplines, and many other fields.

Since it was created as the National Bureau of Standards in 1901, NIST has been a partner with other federal agencies, industry, and universities. In MSEL, cooperation is flourishing. In 1989, 375 visiting scientists and engineers conducted studies with Laboratory researchers or used MSEL's specialized equipment, some of it unavailable anywhere else in the United States.

Our specialized facilities include an array of metals and ceramics laboratories for controlled materials synthesis and process; a 12-million-pound test rig for evaluating large-scale mechanical material properties; experimental stations for studying arc-welding processes; specialized small-angle and texture x-ray diffractometers particularly suited for polymer characterization; and a 20-megawatt reactor for neutron-scattering experiments.

Soon this arsenal of research equipment will feature the nation's first dedicated facility for "cold neutron" studies, filling a serious void in the nation's materials science and engineering research. The Laboratory's Cold Neutron Research Facility (CNRF) will provide beams of deeply penetrating low-energy neutrons, essential for important experiments that are impractical or even impossible with conventional neutron sources.

The building for the Cold Neutron Research Facility was dedicated in January 1989, with the first experimental stations becoming available later in the year. By 1992, it will house 15 experimental stations, including 5 that will be instrumented and operated by outside groups.

Five instruments are now being developed, including a high-resolution small-angle neutron scattering spectrometer, a joint project of NIST and Exxon Research and Engineering. The National Science Foundation and NIST will jointly fund the development of a Center for High Resolution Neutron Scattering at the CNRF. The new facility, which will be available to all U.S. users, is being designed to exploit the most recent advances in technology, resulting in research capabilities that exceed those of cold neutron sources in other countries.

A National Resource

MSEL's research programs and services are crafted according to the needs of U.S. industry. Obviously, we cannot tackle all of the many important issues that must be resolved before industry can fully achieve the promise of advanced materials and manufacturing methods. But, in many instances, we are uniquely qualified to address a certain need.

The Laboratory's data centers, for example, have stepped up their efforts to make vital, critically evaluated information—such as phase diagrams and corrosion-performance data—available to industry and the scientific community. The centers are also developing computer programs for searching materials databases and for applying data to specific problems.

Through conferences, workshops, and our many cooperative programs with industry, we have identified key areas where our research activities can make important contributions. The Laboratory's advanced ceramics program, begun in 1985 and now reaching



full strength in both staff and facilities, is one example; a second is our high-performance composites project, which emphasizes research relevant to high-volume, low-cost applications such as vehicles. Yet another example is MSEL's participation in the NIST-wide initiative to help U.S. industry overcome the many formidable obstacles that stand in the way of commercializing the new high-temperature superconductors.

Take a moment to review the research accomplishments highlighted here. Though not a comprehensive listing, these examples help explain who we are, what we do, and how we pursue some of the many opportunities unfolding in materials science and engineering. And keep in mind that the Laboratory, like all of NIST, is a national resource, in terms of its scientific and engineering expertise and its facilities. I am confident that, upon reading these pages, you will identify ways to make beneficial use of this resource.

A handwritten signature in cursive script that reads "Lyle H. Schwartz".

Lyle H. Schwartz
Director, NIST Materials Science
and Engineering Laboratory

Metallurgy Division

Responding to the international emphasis on high-technology, high-value-added materials, the Metallurgy Division has mounted major research programs on rapidly solidified metals, intermetallic alloys, metal-matrix composites, and nanocrystalline materials. The radically different structures of these novel materials present tremendous opportunities for improving structural, mechanical, electronic, and magnetic properties—the foundation for technological advances. Consistent with the division's long-standing mission, research focuses on characterizing the materials according to their fundamental physical and chemical features and on developing methods to measure these features and the resultant material properties.

The promise of advanced metals will go unfulfilled if the materials cannot be manufactured economically and reliably. In tandem with its basic research programs, the division works to optimize processing methods, an effort that encompasses manufacturing of more conventional alloys. With the aim of improving production processes and product quality, division researchers are applying their measurement expertise to the development of automated, on-line process-control sensors. In turn, this activity meshes with process modeling studies that are identifying the key manufacturing parameters that must be measured and controlled. Modeling efforts are concentrating on powder processing because it offers the dual advantages of near net-shape fabrication and the ability to tailor microstructure to achieve superior material properties. Conducted in cooperation with the NIST Office of Nondestructive Evaluation, current studies address both droplet atomization and powder consolidation.

The division also generates, evaluates, and compiles materials data. It has developed two new databases—one for phase

diagrams and the other for evaluating corrosion performance.

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Powder Processing

A 1986 study commissioned by the U.S. Air Force estimated that net-shape fabrication could reduce the total cost of metal parts in military and civilian aircraft by as much as 90 percent. This finding testifies to the great potential of net-shape technology. Key to achieving this potential, however, are improvements in powder processing, one of the division's objectives.

Division scientists have developed a supersonic inert gas metal atomization (SIGMA) system that produces ultrafine (<50 micrometers in diameter) metal alloy powders. The new system permits carefully controlled studies of the effect of processing parameters on powder size and shape. A holographic laser diffraction measuring system tracks the break-up of the molten metal stream and the formation of ultrafine solid metal powders. An on-line, high-speed photographic system provides real-time measurement of particle size, permitting feedback control of the powder-formation process.

Division studies of tin-, aluminum-, and copper-based alloys have shown that particle size is a function of the gas-jet velocity, liquid-metal flow rate, and composition of the metal powder. A consortium of industrial firms, including Crucible Materials Inc., the General Electric Corp., and Hoeganaes, have joined in this research program.

Consolidation of metal powders by hot isostatic pressing (HIPing) also is being investigated. Division scientists have developed an eddy-current sensor to determine changes in particle density during HIPing. The technique is being used to test theoretical models of

the sintering and compaction processes. Eventually, the sensor will permit real-time feedback control of powder consolidation.

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Rapidly Solidified Metals

Rapid solidification processing (RSP) has spawned a fertile area of metallurgical research. The technique has been used to create alloy systems and compositional regimes that were not even conceivable with conventional processing methods. Suppression of equilibrium reactions allows less stable—sometimes exotic—phases to persist, creating, as one scientist has described it, a "netherworld of stability" from which "surprises will continue to arise."

Aluminum alloys containing large additions (10 percent) of elements with limited equilibrium solubility (<2 percent) are one new class of alloys made possible by RSP. Instead of freezing with large, brittle intermetallic phases, these alloys can be processed to contain a fine dispersion (50 nanometers) of the intermetallic phase. The slow microstructural coarsening rate results in improved high-temperature (340 °C) performance. Division scientists have concentrated on Al-Fe-Si alloys. More than 20 stable and metastable ferrosilicide phases have been reported for this system. Rapidly solidified alloys, however, contain only two phases in combination with the primary aluminum phase: a bcc phase, designated alpha-(Al, Fe, Si), that is very resistant to coarsening and an amorphous phase with extended short-range order. Electron microscopy studies suggest that the relationship between the structures of the two phases is akin to packings of Mackay icosahedra, the units often used in models of quasicrystal phases. During heat



treatment and consolidation of powders, the relationship appears to control the nucleation of the bcc phase. Consequently, the relationship between the phases is directly related to microstructural changes during consolidation.

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Process Control

Measurements of key processing parameters are essential to improving processing efficiency and product reliability. Data gathering, however, is not enough. Information must be immediately available and, then, analyzed quickly to pinpoint necessary processing changes that are made in equally rapid fashion. The ideal, of course, is automated process control, a goal the division is pursuing in its work on on-line sensors and process modeling. One example is the previously cited project on powder processing and HIPing. Another example is described below.

The effects of atomization conditions on the average particle size of metal powders are being measured in the Laboratory's powder processing laboratory.

In collaboration with the Aluminum Association and the NIST Office of Nondestructive Evaluation, the division has developed noncontacting eddy-current sensors for measuring the internal temperature of aluminum rods, tubes, I-beams, and other products during extrusion processing. The real-time measuring technique promises to improve the quality of finished extrusions and to reduce waste caused by processing errors. An important step toward an automated process-control system, the sensor has been tested at commercial aluminum extrusion mills. Temperature readings, taken at the rate of about one per second, were accurate to within 5 °C.

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Data Programs

Like the materials they describe, data on the properties of metals are valuable commodities—for design, processing, and service-performance analyses. The division houses two data centers that serve users from industry, government, and universities.

■ In collaboration with ASM International, the Alloy Phase Diagram Center carries out a series of data evaluation projects. Coverage of alloy systems includes crystal structure, metastable and constrained equilibria, and high-temperature thermodynamic properties, as well as stable equilibrium diagrams. In 1987, the center published *Binary Alloy Phase Diagrams*, a two-volume compendium containing 1,578 phase diagrams. A second edition is planned for 1989.

The center is nearing completion of a relational database of phase diagrams of binary metal alloys. To be operated by ASM International, the database now contains about 1,600 binary phase diagrams, which include 9,000

phases and 7,300 invariant reactions.

■ Operated jointly by the division and the National Association of Corrosion Engineers (NACE), the Corrosion Data Center provides critically evaluated data on the corrosion performance of engineering materials. PC-based computer programs, using standardized formats, have been developed using relational database and expert system technology to provide data for metals and nonmetals in a broad range of industrial environments. Programs access both kinetic and thermodynamic data to aid in the selection of optimum materials and corrosion control systems.

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Magnetic Materials

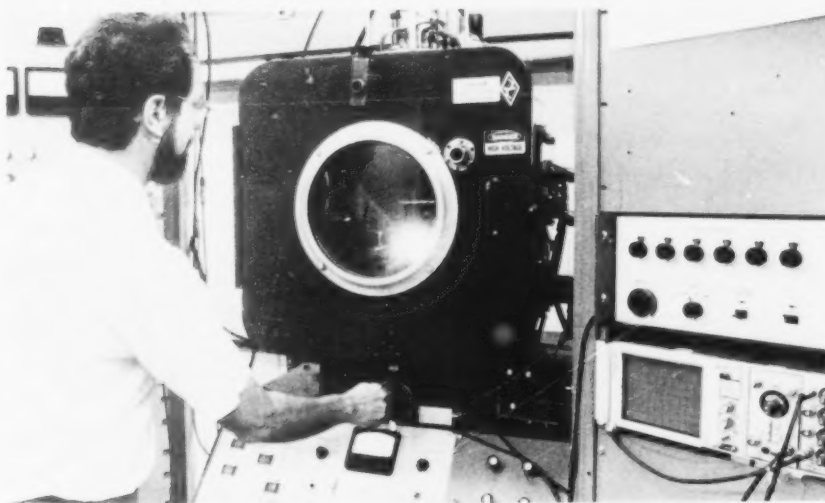
Magnetic materials are of significant technological and economic importance. In the United States alone, annual

sales of soft magnetic materials—primarily for information storage—are estimated to exceed \$25 billion, and sales of hard magnetic materials—primarily for electric motors—top \$1 billion. The division's research on magnetic materials, which includes studies of superconductors, focuses on relating material structures to metallurgical, magnetic, and electronic properties. Part of the research effort is devoted to developing and assessing magnetic methods of nondestructive evaluation of materials.

■ In a research first, division scientists produced a granular-metal thin film of an immiscible mixture of nanometer-sized particles of a magnetic oxide (Fe_3O_4) and a nonmagnetic metal (Ag). Produced by cosputtering, the thin film exhibits several unusual magnetic properties, including superparamagnetism, ferromagnetism, and percolation effects.

■ Division scientists and a colleague from Johns Hopkins University used a novel low-temperature laser-ablation process to produce, directly from the vapor phase, thin films of the new high-temperature superconductors. The critical temperatures of the films were equal to those of bulk specimens, which are created in a high-temperature process. In a film of Bi-Sr-Ca-Cu-O, the Meissner effect was detected at 110 K. The superconductivity of this material was detected with a new technique, magnetic-field-modulated microwave absorption.

■ Division-developed composition-modulated alloys, which consist of thin alternating layers of different metals or alloys, exhibit unusual magnetic properties. For example, alloys of alternating copper and nickel layers display time-dependent magnetization, a behavior attributed to the nickel. This behavior, however, is contrary to expectations. Normally, nickel relaxes very rapidly, but in the new material, magnetization decays



The measurement of process parameters during the rapid solidification of metals is being carried out in the metallurgical processing laboratory.

slowly after the applied magnetic field is removed.

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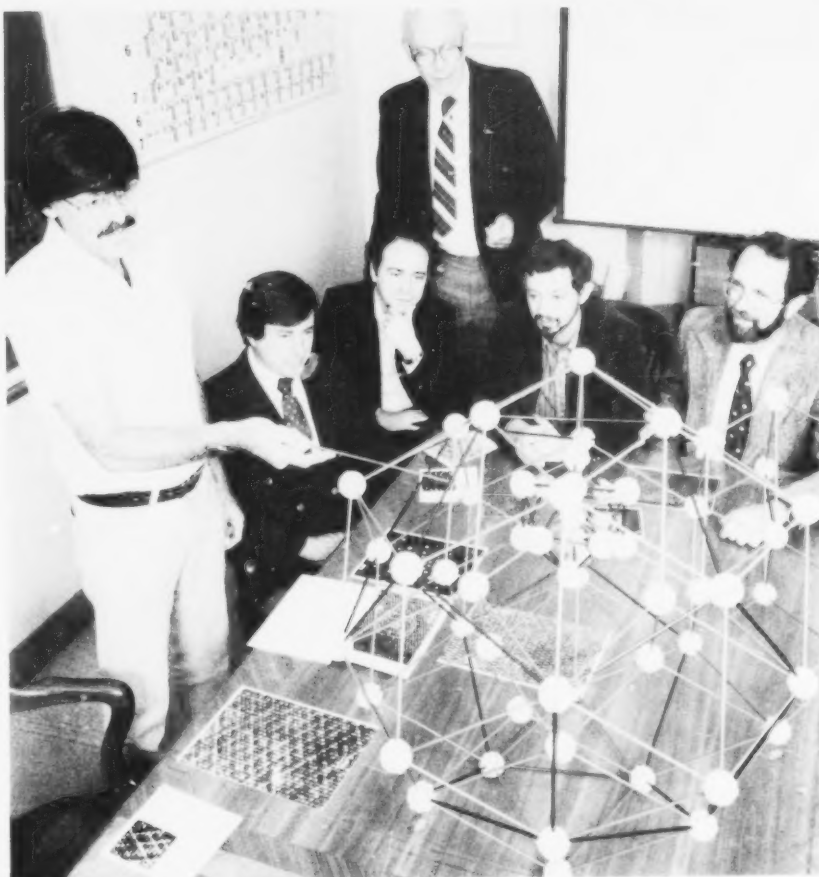
Electrodeposition

Electrodeposition is a ubiquitous manufacturing process. It is used in the fabrication of galvanized-metal sheets and wires, the application of corrosion-resistant coatings, and the production of compact discs, hard disk drives, and other magnetic materials. Aims of the division's research on electrodeposition include identifying, measuring, and modeling important materials and processing variables that affect the properties of coating and developing new approaches to create novel materials.

The process of electrodepositing metal alloys on a moving substrate has been modeled mathematically. The model can be used to predict the composition of the deposited coating or the composition gradient. Intended to serve as the basis for a real-time feedback control system, the model is now being evaluated in an experimental electrodeposition plating cell, which permits control of all significant processing variables.

Division scientists have achieved precision-control over the electrodeposition process, allowing them to apply very thin (about 50 angstroms thick) layers of alloys. The approach is an attractive alternative to molecular beam epitaxy for fabricating compositionally modulated alloys, or superlattices. Materials developed with the electrodeposition method are now being characterized.

The Achilles' heel of composite materials are structural weaknesses that occur at the interfaces between reinforcing fibers and the



matrix. Division scientists are using the precision-control electrodeposition process to coat the continuous fibers in metal-matrix composites. One aspect of this work is to apply coatings with a gradually changing composition to promote bonding between the fiber and matrix. Besides enhancing chemical bonding, the coatings could reduce the high temperatures now required for hot pressing the fibers and matrix into a composite structure, thereby minimizing the potential for damaging reactions.

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A team of NIST scientists and guest researchers led by John Cahn discuss quasicrystal structures in alloys. The discovery of quasicrystal structures in materials challenges a 100-year-old theory of crystallography and the existence of quasicrystals may make it possible to produce materials with radically different properties.

Ceramics Division



Most nations vying for technological leadership in the future world economy have identified the development of advanced ceramics as critical to their success. In structural uses, these materials offer the advantages of high specific strength and high resistance to wear and corrosion. In electronic applications, advanced ceramics already play an important role in the manufacture of semiconductors, transducers, sensors, optical devices, and motors.

Brittleness and lack of reliability, however, are major problems, particularly in high-performance,

Crack growth from indentation flaws are being recorded on video tape so that the qualitative aspects of the intrinsic fracture resistance of ceramics can be documented.

structural applications. The Ceramics Division, in collaboration with other NIST units and outside organizations, has mounted a major program to develop the basic standards and technology necessary for advanced processing of ceramic materials. In addition to projects addressing the composition, structure, and properties of

ceramics, the program includes studies relating to processing science, ceramic powder characterization, tribology, and ceramic matrix composites. With the discovery of ceramic materials that are superconducting at high temperatures, division researchers have been developing the basic data required for processing this unique class of materials, and they have measured many of the new superconductors' physical properties.

To facilitate its research, the division has built state-of-the-art clean-room facilities that permit rigorously controlled studies of ceramics processing. Our new surface forces laboratory is now being used for research on phenomena critical to understanding fracture, sintering, and tribology.

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Models

Common to the division's many research projects on advanced ceramics is the objective of developing the theoretical framework to guide improvements in ceramics processing and in the materials themselves.

Division scientists have developed a theory that explains why fracture toughness increases with crack size in monophase ceramics. In recently completed work, they used indentation flaw techniques to study strength behavior in a number of ceramic materials. Crack growth was videotaped, permitting qualitative assessment of aspects of fracture resistance. The results suggest that interlocking grains and ligaments of unbroken material in the wake of the crack front apply a closure force to the crack surface; for the crack to propagate, it must overcome this force. Derived from these observations, a division-developed model predicts strength as a function of

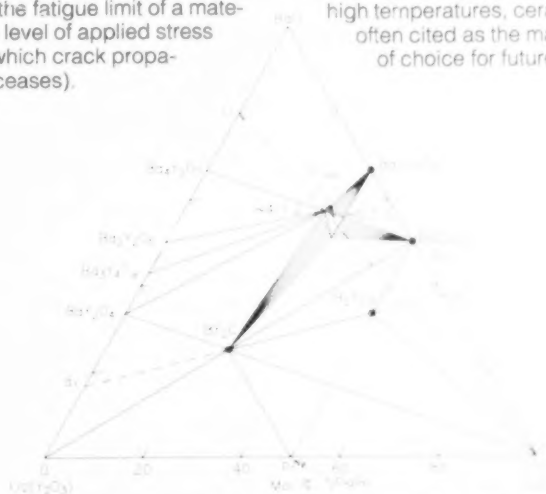
microstructure. The model has the potential to guide the microstructural tailoring required to achieve optimal toughness in ceramics. With researchers from Lehigh University, division scientists are evaluating the model's utility. They are fabricating monophase "composites" with duplex grain structures that should provide maximum resistance to crack growth.

The role of surface forces in subcritical crack growth also is being investigated. Division scientists have found that, in addition to cohesive covalent and ionic forces, there are weaker, long-range adhesive forces operating when new surfaces are created. These include dispersion (van der Waals), electric double layer, solvation, and cation-site forces, all of which play important roles in colloidal chemistry. Fracture experiments on mica, glass, and sapphire demonstrated that the forces also shape the strength properties of ceramics, participating in the reversal of crack growth (crack healing) and partially determining the fatigue limit of a material (the level of applied stress below which crack propagation ceases).

Determination of the relationship between surface closure forces and crack properties is leading to a fundamentally new concept of fracture. According to this concept, the underlying process of bond rupture at the crack tip operates independently of the adhesive forces behind the tip. The decoupling of the two processes provides the impetus for atomic-level investigation of crack-tip structures. More important, the research suggests that fracture can be used to measure surface forces. Using data from studies of mica and other brittle ceramics, division scientists have developed a theoretical model that relates fracture behavior to surface force functions, which can be measured independently.

The models developed by division researchers include several that predict phenomena that occur during sintering, as well as others that account for physical and chemical processes at matrix inclusion interfaces in composites.

Because of their tolerance for high temperatures, ceramics are often cited as the material of choice for future engines.



Phase diagram of the $\text{BaO-Y}_2\text{O}_3\text{-CuO}$ system determined experimentally in the Ceramics Division. These data were used by researchers at NIST and elsewhere to prepare specimens of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ compound recently shown to be superconducting at temperatures near 95 K.



The interrelations between processing, microstructures, and properties are being determined for the new high-transition temperature ceramic superconductors. One of these superconductors is shown being removed from the furnace.

offering better performance and high fuel efficiency. To help U.S. industry achieve this goal, the division conducted a comprehensive assessment of the major research issues that must be addressed. After a review of the technical literature and consultations with representatives of industry, division scientists concluded that data are needed in four general areas: performance, wear prediction, surface reactivity, and surface forces.

With this guidance, the division has initiated a research program to define the limits and requirements of ceramics under various environmental conditions, speeds, loads, and temperatures. The resultant data are expected to lead to detailed understanding of the mechanisms of wear in advanced materials and, ultimately, predictive models of wear.

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Processing

The division's activities in ceramics processing encompass research on powder characterization, processing chemistry, sintering, microstructural properties, and the influence of processing variables on material properties. Many of the projects in this area will benefit from the division's ultra-clean (an extrapolated cleanliness of Class 10) ceramic processing facility. Production of ceramic powders with controlled impurity levels is now possible, permitting detailed study of the influence of chemical composition on sintering. In division studies, the effects of contaminated powders, with predetermined concentrations of impurities, will be evaluated during various processing steps, such as synthesis, compaction, calcination, and sintering.

Because starting powders are highly variable ensembles of particulates, industry and researchers need Standard Reference Materials with certified distributions of particle sizes to calibrate measurement instruments. The division has developed a silicon powder Standard Reference Material for calibrating x-ray line positions in x-ray powder diffraction. It is also play-

ing a major role in an international "round robin" exercise to assess the powder-characterization capabilities of research organizations and ceramics manufacturers. The division has prepared about 1,000 samples of zirconia powder and about 3,500 samples of silicon nitride powder, all of uniform physical and chemical composition. Evaluation of the results of the round robin, which was sponsored by the International Energy Agency, is now under way.

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Data Activities

Assessments of the status and needs of the U.S. manufacturing sector often point to the shortage of data on the physical properties of many materials. A 1986 National Research Council study, for example, cited this lack of basic information as "a severe handicap in manufacturing process development."

The Ceramics Division has several programs that aim to provide design engineers and research scientists with timely compilations of critically evaluated data. Recently, the division began efforts to develop a national database system on the properties of ceramics. Examples of ongoing data activities are described below.

■ During the last few years, the division and the American Ceramic Society (ACerS) have greatly expanded their long-standing cooperative program of compiling and disseminating phase-diagram data. Industrial contributions of more than \$2 million helped support expansion of the program, indicating industry's growing need for broader, more up-to-date coverage of evaluated phase-diagram data for ceramics.

The program published the sixth volume of *Phase Diagrams*

for *Ceramists* in late 1987. Produced with the program's newly developed computer graphics, database, and typesetting systems, the publication contains 697 commentaries and 1,080 binary, ternary, and higher-order phase diagrams of oxide systems. In addition, program-developed graphics and search software for personal computers is now in the testing and development stage, an important step toward making phase-diagram data available on-line. Other programs for manipulating data for research purposes are being evaluated.

■ Research on tribology—the study of friction, lubrication, and wear of interacting surfaces that are in relative motion—is highly interdisciplinary and, as a result, sometimes fragmented. The results of tribology studies are published in many different journals, depending on the specialty of the researcher. Consequently, progress in the field is often hindered by the inaccessibility of critical data and the lack of communication between researchers with complementary interests but different disciplinary affiliations. To overcome this problem and encourage technology transfer, the division, several federal agencies, and two professional societies have embarked on an international project to develop a computerized tribology information system (ACTIS). Recently, a prototype of ACTIS was successfully demonstrated.

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Superconductors and Optical Materials

Complementing the division's research on mechanical properties of ceramics are studies of the materials' optical and electrical properties, which are of interest to semiconductor manufacturers and other industries. An example is the

division's program on ceramics for optical applications, which focuses on thin films. Specifically, this program addresses how processing parameters affect the structure and performance of diamond and other ceramic coatings.

Work on the new high-temperature metal-oxide superconductors spans the division and the entire Institute. The division's participation in the Institute-wide initiative to support industry's efforts to achieve the technological promise of these remarkable materials addresses issues related to processing.

■ Division researchers have developed a relatively simple method for depositing diamond films. Verified by a variety of analytical techniques, including Raman and Auger electron spectroscopy, the surface application was achieved with hot-filament chemical vapor deposition. To date, diamond films have been deposited on silicon, silica, and silicon carbide. Wear tests indicate that the films are extremely hard and tenacious. Alumina, silicon carbide, and even a diamond stylus abrade when rubbed against the films. Stylus measurements indicate that the film is rough, but polishing can smooth the surface. The films transmit well in the infrared at wavelengths longer than 10 micrometers. At shorter wavelengths, however, optical scatter results because of the films' roughness.

■ Shortly after the announcement in early 1987 that the compound $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ was superconducting at temperatures near 95 K, division researchers determined the phase equilibria for the $\text{BaO}-\text{Y}_2\text{O}_3-\text{CuO}$ ternary system and the three included binary systems. NIST researchers and others used these valuable data to prepare single-crystal and polycrystalline specimens to examine the superconductor's properties. Division studies showed that superconducting properties such as transition temperature and transition width, as determined by measurements

of electrical conductivity and AC magnetic susceptibility, are very sensitive to annealing temperature and sintered density. Compositional mapping proved to be an accurate method for assessing the homogeneity of microstructure.

Experiments aimed at characterizing the fracture behavior of the superconductor were also performed. Fracture strength and fracture toughness were measured on samples sintered at 960 °C in either air or oxygen and annealed at 600 °C in either air or oxygen. The sintering and annealing environments were shown to affect both properties, with the highest strength and toughness observed in as-sintered material fired in oxygen. Annealing caused both the strength and toughness to drop considerably, indicating that processing conditions affect the intrinsic properties of the material, not just the flaw population. In addition, division studies showed that moisture enhances crack growth in these materials, as it does in most other ceramics.

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Polymers Division

The uses of polymers and polymer-based composites are virtually unlimited, and the materials are even displacing metals in many structural and high-performance applications, including automobiles and commercial and military aircraft. Advances in polymer science have paced important technological developments. The most dramatic example, perhaps, is the silicon chip, which owes its intricate electronic architecture to the polymeric photoresists used during the chip-etching process.

The Polymers Division, with a research staff of about 60, supports U.S. industries that produce, process, or use synthetic polymers. Its basic research programs are devoted to strengthening the scientific foundation that sustains continued advances in this important class of materials. These programs are designed according to the perceived needs of industry. The division's increased emphasis on detailing the chemical and physical processes that underlie phase separation in block polymers is one direct response to a critical need identified by industry and the scientific community.

Another is the division's comprehensive program on polymer composites, which addresses issues related to the processing and durability of these materials.

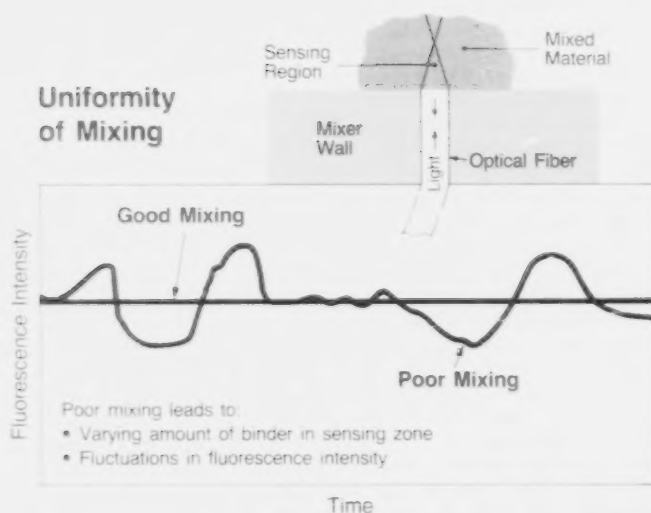
Even though the United States produces more than 50 billion pounds of polymers annually, design and manufacturing processes can be improved substantially. Polymer processing is now guided largely by empirical methods. Understanding of the fundamental principles of processing is needed. To help build this science base, the division is developing novel sensors for monitoring viscosity, flow, molecular orientation, and mixing.

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Specialty Polymers

In one of the division's long-range research programs, scientists are investigating the interaction of electrical fields with polymers. Their aim is to improve existing measurements and develop new ones, as well as synthesize new polymers for special applications. Studies address such topics as charge or polarization distribution in thin films, piezoelectric and pyroelectric polymers, and electro-optical properties. An example follows.

Division scientists have developed an improved thermal pulse technique to determine the charge distribution in dielectric polymers. Pulsed laser beams generate a temperature gradient, permitting measurement of charge flow during temperature equilibration. These measurements can be mathematically related to the initial space-charge distribution. Such information contributes to the understanding of charge injection during



A schematic illustrating a new technique that uses fluorescent probes to monitor important processing variables. By measuring changes in the fluorescent spectra of organic dyes, sensors can determine the degree of mixing, melt viscosity, temperature, and molecular orientation of polymers.

voltage stress and its influence on electrical breakdown phenomena in polymer dielectrics.

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Polymer Standards

The division develops measurement techniques and Standard Reference Materials for the polymer industry. The emphasis is on providing tools that aid in predicting the processability, properties, and service lifetimes of polymers. In support of this activity, the division conducts research on the properties of polymer solutions and on the mechanisms of polymer degradation.

■ Gel permeation chromatographs are the polymer industry's primary means of measuring molecular weight and molecular weight distribution, important indicators of end-use properties. The division is developing two polyurethane Standard Reference Materials for calibrating chromatographs. Polyurethanes are used widely in implanted medical devices, but the composition of the block copolymers vary widely. The Standard Reference Materials should improve characterization of these important materials.

■ Two poly(methyl methacrylate) Standard Reference Materials have been certified as molecular-weight standards. They will be used by the Navy for quality control of antifouling paints and will find general use as calibrating materials for the analysis of acrylic polymers. A polyethylene Standard Reference Material has also been issued as a melt user standard, in response to user requests.

■ Division scientists have devised techniques that use fluorescent probes to monitor important processing variables. By measuring changes in the fluorescent spectra of organic dyes, the sensors can

determine the degree of mixing, melt viscosity, temperature, and molecular orientation. Components of the system are optical fibers, which irradiate the sample and collect the emitted fluorescent light, and fluorescent chromophores, which are dispersed in the polymer melt. Division scientists have used a similar approach to monitor the formation of polyimides from poly(amide-acid) precursors. This method can be used for quality control in the manufacture of electronic packaging materials.

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Mechanical Performance

Analyses of the structure and mechanical behavior of polymers are essential to efforts to improve the performance of the materials and to extend their applications. Scanning tunneling electron microscopy is being adapted to characterize polymer structure at the molecular level. Information gained by STM bridges the size scales of conventional electron microscopy and spectroscopic techniques. Research on mechanical behavior focuses on stress relaxation, creep, mechanical deformation, and physical aging of polymer glasses.

■ Division studies are detailing the processes underlying the development of spherulitic structures in bulk crystallized isotactic polystyrenes (i-PS). This advance stems from the discovery that amyl acetate readily dissolves amorphous i-PS but not crystalline i-PS. Using an electron microscope, scientists examined the morphological characteristics of the excised incipient spherulites at different stages of development. So far, they have learned that profuse interlamellar splaying precedes the degeneration of the lateral growth of lamellae to a fibrillar habit. The intrinsically curved conformations

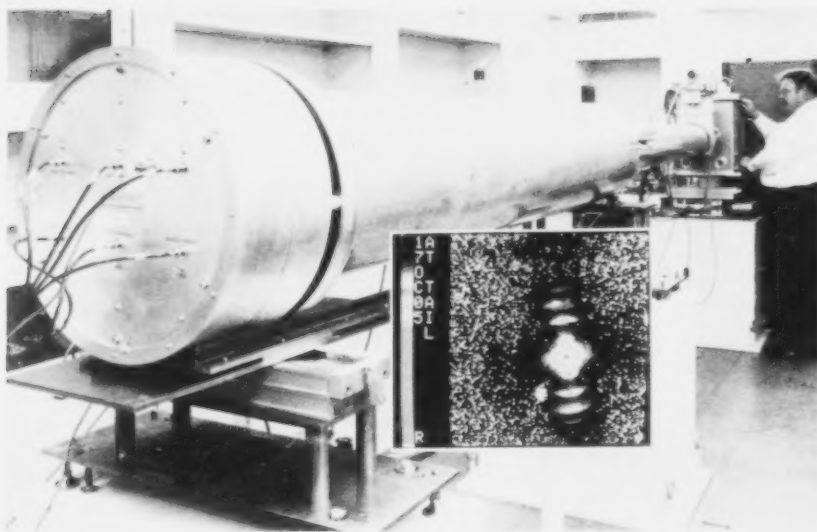
of the lamellae—apparently the result of buckling under the influence of chain folds—seems to cause splaying. Interlamellar links between the fold surfaces of overlapping lamellae were observed, suggesting that splaying is not mainly the result of impurity segregation.

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Composites and Blends

Polymer-based composites and polymer blends present the opportunity to develop materials that are tailor-made for specific applications. Achieving the full potential of polymer composites, however, will require more economical and more reliable processing methods and much more accurate techniques for predicting performance, especially long-term durability. Polymer blends already account for about 15 to 20 percent of plastic materials produced annually in the United States, but most blends on the market are the products of intuition, rather than objective understanding. The division has two major programs that address important issues in research on polymer blends and composites.

■ The properties of composite resins, and hence composites themselves, depend not only on the formulation of the resin but also the microstructure introduced during processing. New measurement methods have been developed to use small-angle x-ray and neutron scattering to evaluate this microstructure. Experiments have shown that variations in processing cycles can significantly increase the fracture energy of a resin. The corresponding variations in microstructure involve differences in the molecular network structure of the resin. The changes can involve



The small-angle x-ray scattering facility is used by polymer physicists to obtain information on the microstructures of polymer blends to develop polymer alloy phase diagrams. Inset shows x-ray scattering pattern.

variations in the cross-link density of the resin and/or differences in the heterogeneity of the cross-linking at the same average cross-link density. Bulk properties, such as the glass transition temperature, depend on average cross-link density. Consequently, changing the heterogeneity without changing the average structure provides a potential approach to increasing toughness without loss of bulk properties. Current studies are investigating this approach.

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The dynamics of concentration fluctuations in a deuterated polystyrene/poly(vinylmethylether) blend system has been studied with temperature jump and reverse quench light scattering techniques. The linear theory of Cahn-Hilliard-Cook for spinodal decomposition has been verified quantitatively for the first time with this mean-field

polymer/polymer blend system. Together, with data from SANS measurements, derivatives of free energy, interdiffusion coefficient and interfacial free energy density have also been obtained. The interdiffusions coefficient is continuous at the phase boundary, and the mobility has an Arrhenius type of temperature dependence on both sides of the boundary.

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Bioprocessing

Division scientists have investigated desulfurization of coal by extremely thermophilic bacteria. They have developed procedures for routine cultivation of these bacteria at 99 °C and determined the range of substrates from which sulfur is removed by these bacteria. They have also initiated testing polymers

for their biodegradability, in response to the emerging need for the definitions of the test methods for plastics biodegradability. In addition, standards for metal ore bio-leaching, including testing procedures and reference materials, are currently under development.

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Dental and Medical Materials

Dental and medical materials must perform reliably in the chemical environment of the human body. If a material fails, the consequences can range from physical discomfort to death. One of the division's research programs seeks to develop materials and techniques that promote adhesion to teeth, dentin, enamel, bones, and soft tissues.

When placed in the mouth, resin-based dental composites shrink during polymerization. This either lowers adhesion to the tooth or leaves a gap that is susceptible to caries (decay). Division scientists are working on new monomers that expand on polymerization and can be used in dental composites. Preliminary studies show reduced shrinkage and improved adhesion can be obtained in the laboratory. Efforts are also in progress to improve the degree of cure as undercured composites are less durable. An acrylate monomer that forms a cyclic chain during polymerization has been found capable of attaining nearly 100 percent cures at in-mouth temperatures. The development of composites employing monomers of the types described hold promise for more durable, disease inhibiting restorations in the future.

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Materials Reliability Division

Understanding how and why structural materials fail—the aim of the Materials Reliability Division—can yield enormous benefits. When used in the design and fabrication of structure, detailed, quantitative knowledge of the mechanics of fracture and deformation can improve safety and reliability, increase productivity, and even avert disaster, such as bridge collapses and railroad derailments that stem from stresses and flaws in materials.

With about 30 members, the division's interdisciplinary research staff studies nonlinear fracture mechanics, arc physics, acoustoelasticity, the mechanics of composite materials, and the relationship between the structure, properties, and mechanical behavior of materials. Examined over a wide range of temperatures, from ambient to cryogenic, the materials of primary interest are metals and alloys, advanced composites, and welds. The products of these efforts include improved fitness-for-service criteria, quality-control sensors, and techniques to improve the deformation processing of aluminum and high-strength steels.

In addition to NIST-sponsored research, the division conducts studies for other government agencies and provides technical services to industry and public and private research institutions. Programs sponsored by other federal agencies are primarily related to the safety of structures, pressure vessels and piping, transportation equipment, and cryogenic systems. Collaborations with industry range from analyses of new alloys to the testing of more than 1,000 weld specimens as part of an effort to develop nationally recognized welding procedures.

Besides the expertise of its staff, the division offers modern laboratories, which feature servohydraulic fatigue- and impact-testing machines for fracture-mechanics research; electromag-

netic and radiographic equipment for nondestructive evaluation; experimental stations for arc-welding processes; and instrumentation for measuring mechanical and physical properties, especially at cryogenic temperatures. Other research tools include facilities for computer interactive testing and for x-ray diffraction, metallographic, and mechanical studies.

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Fracture Mechanics

Fracture-mechanics research provides the basis for determining the limits of a structural material's performance and, consequently, for setting safety requirements—allowable stress levels, minimum service temperatures, toughness requirements, and weld-quality standards. For many materials, however, these requirements are derived from empirical methods and experience. A goal of the division is to replace this qualitative approach with the more precise, quantitative methods of fracture mechanics. Such objective tools are especially needed for the design and fabrication of advanced structures, because limited experience offers little or no guidance for assessing safety requirements. Examples of the division's fracture-mechanics research follow.

■ As part of an effort to model the fracture mechanics of weldments, division researchers studied the crack-driving force in tensile panels under three weld conditions: matching strength, undermatching by 20 percent, and overmatching by 20 percent. The study found that overmatching welds significantly deter crack propagation in panels with small cracks. Standard

fracture toughness tests, in contrast, use specimens with deep cracks and, therefore, overlook the benefits that result from welds that exceed the strength of panels.

■ In the temperature range where steels become brittle, fracture toughness values vary considerably, complicating efforts to assess structural safety. Results of a division study account for this wide variation. The research showed that cleavage fracture initiates at a single site located within the region of maximum stress ahead of the crack tip. Four kinds of initiation sites were identified: cleavage facets, unfractured inclusions, inclusion clusters, and local zones of ductile tearing. In the ductile-to-brittle transition region, toughness values varied according to the distance between the crack tip and the cleavage initiation site.

■ The division's fracture-physics research probes the toughness of materials from the perspectives of their fundamental properties, the interactions between cracks and dislocations, and the effects of environmental conditions. Products of these efforts include a model of the behavior of dislocations emanating from sharp cracks and of the effect of dislocation shielding on material toughness. These models were initially developed for metals, and now we are extending them to understand the fracture behavior of composites and dislocation generation in electronic materials.

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Advanced Composites

The division is addressing three areas that are essential to achieving the great potential of composite materials for structural applications. Using acoustical and optical techniques, researchers are investigating the

mechanisms of fracture and deformation in composite panels. In support of the Institute's nondestructive evaluation program, division scientists are using finite-element and finite-difference techniques to model acoustic-wave propagation and electromagnetic fields in composites. Another effort is refining theoretical models and associated computer programs for predicting the properties of composites as a function of the properties, volume fractions, and phase geometries of materials that make up the matrix and reinforcing elements.

■ In laminated composites, damage often begins at the edges where the strains are greatest. Theoretical models have been developed to study interfacial cracks using Green's functions. The models predict the significance of damage (ply cracks, delaminations, edge damage) on fracture strength.

■ Division scientists used an optical-diffraction strain measurement system to determine in-plane, plane stress components of a crack area in composite panels under load. The measurement technique showed the localized high-strain fields near the crack tip, yielding results that agreed closely with predictions made with a finite-element model.

■ Fiber-reinforced composites are highly anisotropic: Properties in one direction differ from those in another. Though this complex compositional arrangement is responsible for the superior properties of these materials, it complicates efforts to detect flaws and measure the physical properties of the entire composite. Division researchers are developing arrays of sensors to overcome this obstacle. One array will enable evaluation of in-plane elastic moduli. Similar probes are being investigated for quantitative measurement of fiber layout, fiber-volume fraction, and porosity.

■ A resonance technique was used to measure the internal friction and torsion moduli of a series of uniaxially fiber-reinforced com-

posites. Both types of measurements were evaluated on the basis of wave-scattering theory to assess their usefulness for quantifying the homogeneity of fiber distribution in the composite matrix. Division scientists determined that measuring the torsion modulus is the superior method for detecting and quantifying irregularities in fiber distribution.

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Nondestructive Evaluation

Division scientists have developed nondestructive methods to measure flaws and residual stresses, the sources of the greatest uncertainty in assessments of structural safety. These detection and measurement methods use three types of non-contacting sensors: electromagnetic acoustic transducers (EMAT's), eddy-current probes, and capacitive-array sensors.

■ Two EMAT-based systems will soon be tested in the field. One will measure residual stress in the rims of train wheels; the other will be used for roll-by inspection of the treads in train wheels.

■ Division scientists also are developing EMAT's to monitor aluminum and steel manufacturing. Measurements of ultrasonic velocities revealed the crystalline texture in steel sheet and correlated well with a measure of formability obtained by industrial collaborators.

■ A division-developed capacitive-array probe successfully characterized surface features in bulk dielectric materials. New electronics may allow the probe to monitor sintering of ceramics.

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Cryogenic Materials

Superconducting magnets and other low-temperature systems require structural materials that can withstand brutal cold. The behavior of materials, however, changes significantly under such extreme temperature conditions. In support of low-temperature applications, division scientists develop and characterize metals, alloys, and composites, and they develop testing procedures and standards. In addition, the division evaluates material properties and provides this data to industry, universities, and other government agencies.

■ Mechanical and physical properties are measured at low temperatures. Unique facilities are used to study creep, fatigue, and microstrain in liquid helium (4 K). New understanding of adiabatic conditions during mechanical tests at 4 K assists in the preparation of test standards.

■ Using ultrasonic methods, division scientists measured the elastic constants of the new high-temperature metal oxide superconductors at temperature intervals between 295 K and 4 K. Below and above the critical temperature (T_c), the elastic constants are irregular, and some materials show hysteresis. As the temperature decreases, the lattice softens just above T_c . Below T_c , however, the lattice becomes increasingly stiff as the superconducting phase develops.

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Welding

Because structural failures often originate in welding flaws, the study of welding is integral to structural safety research. Division scientists develop

procedures that reduce the incidence of defects, and they develop weld metals with improved tolerance of defects. A computerized gas-metal arc-welding system produces flaws used in assessing methods of non-destructive evaluation and in studying the relationship between microstructural features and fracture toughness. In tandem with these studies, researchers are developing automated process controls to improve the reliability and quality of welds.

■ As shown in previous NIST studies, inclusions in welds are a major cause of the weakness of weld metals, relative to base metals. To reduce oxide inclusions, members of the division built a shielding-gas enclosure to eliminate the aspiration of air into the nearly pure argon atmosphere. Oxygen in the weld decreased by 50 percent, resulting in 40 percent increase in toughness. Another project seeks to reduce weld inclusions by modifying the deoxidation products in weld pools. Several deoxidizers are now being evaluated.

■ The natural arc frequency in short-circuiting droplet transfer was mapped over a range of current and voltage. Electrical measurements were made with a stable current regulator and compared with visual observations made with a laser backlighting system. The transfer frequency corresponded to the empirically derived optimal weld conditions used in industry. The resulting algorithm will be used in a real-time control system being developed.

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Thermomechanical Processing

The division has built a deformation-processing simulator for in-depth study of thermomechanical processing of steel, with the aim of achieving the optimal combination of strength and toughness. The simulator's features include: vacuum to 1×10^{-5} torr; deformation rate to 20 mm/mm/s; load capacity to 250 kN; cooling rate to 25 K/s; multiple strike capability; and a 450-kHz induction heater.

In studies conducted with the simulator, division scientists determine the optimal temperature, deformation schedules, and cooling conditions for hot rolling and direct forging. In ongoing research, staff members are identifying the metallurgical changes that occur in microalloyed steels during processing. A double-deformation technique is used to measure the recrystallization temperature and to study the influence of the austenite condition on continuous cooling transformation (CCT) kinetics.

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Reactor Radiation Division

A 1977 study by the National Research Council estimated that about 250 researchers nationwide were conducting neutron-scattering experiments. Ten years later, the NIST research reactor alone was host to over 200 scientists and engineers (most for more than one visit), not including the 17 permanent scientists of the Reactor Radiation Division (RRD) and many other NIST staff members. The visiting researchers represented 16 federal agencies, 37 U.S. universities, 23 U.S. industrial firms, and 16 foreign laboratories. More than 60 percent were involved in collaborative studies with division scientists.

The tremendous increase in activity attests to the great research value of neutron-scattering methods—to materials science, biology, physics, chemistry, metrology, and other fields. In materials science, these techniques permit studies of bulk samples, often yielding information on submicroscopic materials behavior and structure that is unsurpassed in detail and accuracy. Small-angle neutron scattering (SANS) is a unique and powerful probe of polymer structures. It can be used, for example, to track processing-caused microstructural changes, such as the emergence of microcracks, over a range from a few nanometers to a micrometer.

Division scientists are engaged in a broad research program aimed at understanding and measuring the structure and properties of virtually all classes of materials used by industry, including high-temperature superconductors, advanced ceramics, catalysts, artificially structured materials, hydrogen in metals, and others that hold promise for high-technology applications. They are also furthering the uses of neutron diffraction and radiography for nondestructive evaluation. Other activities include efforts to develop new

instrumentation and to strengthen the theoretical foundation of neutron-scattering research.

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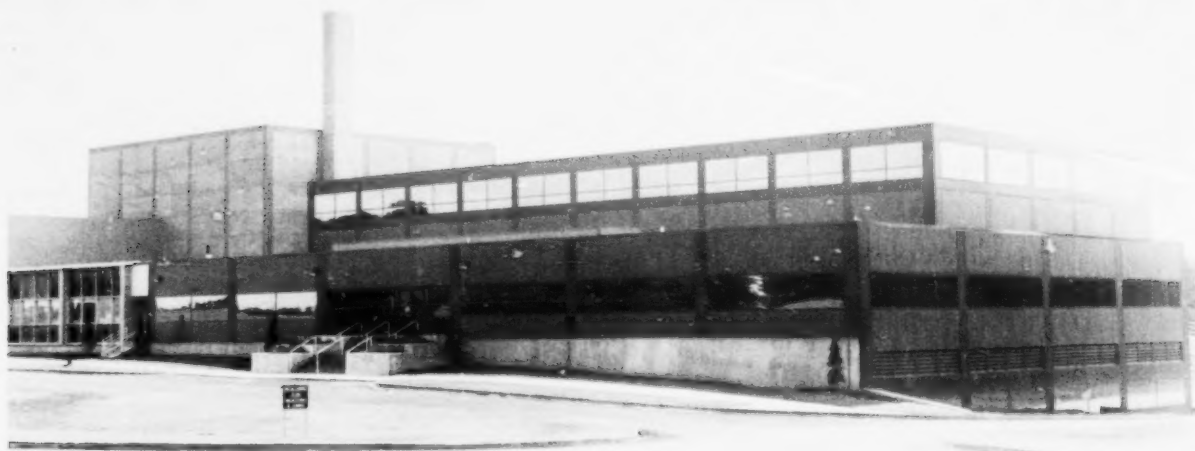
"Cold" Neutron Facility

NIST is constructing the Cold Neutron Research Facility (CNRF), which will greatly enhance research capabilities. Increasing the flux of long-wavelength neutrons (those at temperatures of ≤ 60 K) will permit studies that are not now possible at any facility in the United States. The range of elements that can be detected with neutron depth profiling, for example, will be extended and will include oxygen and nitrogen. Researchers will be able to detect subsurface impurities and dopants in concentrations of less than 1 part per million, with lateral resolution of a few micrometers. SANS experiments will also benefit from substantially increased sensitivity and resolution.

The 20,000-square-foot experimental hall is now completed. To be installed are seven neutron guides, which will direct cold-neutron beams ($15 \times 6 \text{ cm}^2$ in cross-section) to 15 experimental stations, one-third of which will be built by non-NIST organizations. Examples of ongoing work follow.

■ Installation of the neutron guides has begun and on completion in early 1990, the existing time-of-flight spectrometer and the 8-m SANS will be moved from the reactor hall and installed. The 30-m Exxon/NIST SANS, currently under construction, and the neutron-lifetime measurement station, currently being tested, will also be placed on guides, along with the new depth-profiling facility and the prompt-gamma activation analysis station, both of which are being fabricated.

■ NIST and NSF have announced plans to develop a Center for High Resolution Neutron Scattering at



the CNRF. Within 3 years, a 30-m SANS—for the characterization of microstructures in new materials—and a spin-polarized inelastic neutron scattering spectrometer—for the characterization of dynamics and interactions of atoms and molecules, will be completed. Both instruments will have polarized-neutron capabilities for magnetic material studies, and will be state-of-the-art in instrumental resolution.

■ Development of improved guides, focusing techniques, and super-mirrors to enhance neutron intensity continues both in the division, and in collaboration with DOE personnel.

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Materials Characterization

Often in cooperation with industrial and university researchers, division scientists and other NIST staff members are studying new or improved materials for semiconductor circuits, power transformers, automobiles, aircraft, sonar and microwave devices, microbatteries, and magnetic devices. Studies

address all classes of materials—metals, ceramics, composites, and polymers—as well as catalysts and biological structures. Some recent research accomplishments are described below.

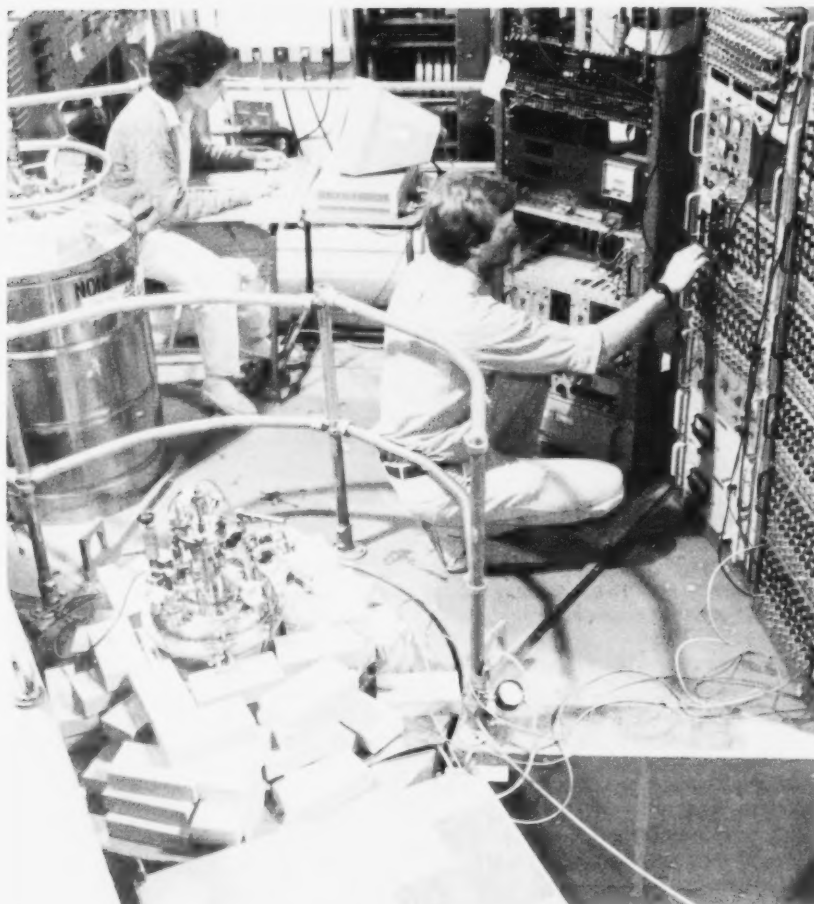
■ The sensitivity of neutron scattering to light elements has made it an indispensable tool in the determination of the atomic structures of the new high T_c superconductors. In addition to complete structural determinations of a number of the newly produced compounds, recent work has also focused on the effects of substituting other atoms for Cu and Y in $YBa_2Cu_3O_{7-x}$. From this work a correlation between valency and preferred site for the substitutions has been developed, as well as a determination that the atomic order in the Cu-O "plane" sites is significantly more critical to high T_c superconductivity than that in the Cu-O "chain" sites. Efforts have also been directed toward examination of the antiferromagnetic order which develops in oxygen deficient materials, or in those for which a magnetic atom has been substituted for Cu or for Y. The antiferromagnetic order can involve both "chain" and "plane" atom sites ordering at different temperatures in the case of the substitutions for Cu, and antiferromagnetic interactions between Y

Cold Neutron Research Facility under construction at the National Institute of Standards and Technology.

sites in the case of magnetic rare earths substituted for Y. This work includes collaborations with scientists from AT&T Bell Laboratories, Bell Communications Research, and several universities.

■ With scientists from the University of Illinois, molecular beam epitaxy methods for rare-earth metals have been applied to the study of artificial metallic superlattices (multilayers) of magnetic rare earths and nonmagnetic Y. Such structures have been shown by neutron scattering to exhibit a long-range coherence of the incommensurate periodic magnetism reflecting a coupling of magnetically "dense" Y layers via the formation of a conduction electron spin-density wave. From these and other results, it has been possible to determine many of the details of the exchange couplings present in the rare earth, and the interplay of the exchange energy with the magneto-elastic and anisotropy interactions.

■ The pairing of hydrogen and metal atoms in rare-earth compounds is highly unusual and



A quasielastic scattering experiment is being set up on the time-of-flight spectrometer to characterize molecular dynamics in a catalytic material.

poorly understood. Studies of high-resolution neutron-vibration spectra for a wide range of hydrogen concentrations dissolved in hcp-yttrium revealed that the pairing is associated with dynamic coupling. This coupling modulates with concentration- and temperature-dependent occupation correlations along the c-axis.

Contrast-variation SANS measurements have determined for the first time the average thickness and degree of uniformity of adsorbate layers chemically bonded to the internal pore surfaces of microporous silica particles. With these

measurements, division scientists are relating the morphology of the bonded phases to trends observed when such bonded-phase particles are used in high-performance reverse-phase liquid chromatography.

Working with researchers from the University of Notre Dame, RRD scientists have established the existence of highly damped propagating magnons in diluted magnetic semiconductors. The materials are examples of highly "frustrated" exchange-coupled systems. In the dilute magnetic-ion limit, neutron scattering has provided a direct measure of the magnitude of the pair-exchange interaction energy. The character of observed transitions has been correlated with theoretical calcula-

tions and with high-field magnetization "step" measurement results.

A joint RRD and Surface Science Division study analyzed the interactions of oxygen and hydrogen with the surface of particles of the catalyst palladium ("cleaned" Pd black). Neutron vibration-spectroscopy indicated that hydrogen atoms are adsorbed on the regular triangular sites on (111) surface planes. Analyses also indicated very strong dynamic interactions between the hydrogen atoms. A lower-energy feature in the spectra suggested that hydrogen bound in subsurface layers at octahedrally coordinated sites was responsible for the strong interaction. Such subsurface species were predicted by theory and can be related to the observed macroscopic behavior of palladium and other metals.

The ability to engineer materials has given rise to an array of unusual combinations of properties, structures, and physical phenomena. Graphite intercalated compounds, an example of artificially layered or modulated structures, have been the object of intense study by division scientists and collaborators from the University of Illinois, Michigan State University, and the Institute Laue-Langevin. The team has been focusing on two-dimensional potassium-ammonia solutions intercalated in graphite. One aim is to explain the structure and orientation of the inserted ammonia and potassium atoms. They have found, for example, that above the melting transition temperature, ammonia molecules and potassium ions form four-fold complexes. In related work, neutron spectroscopic and diffraction studies of pillared clays—with great potential for tailored applications in chemical catalysis and exchange—have provided new insight on the orientation of pillaring ions. Direct information has also been obtained on the torsional potentials of intercalated tetramethyl ions, which provide the first experimental results to test current models of the interlayer template potential of a clay.

■ RRD scientists have discovered the existence of long-range periodic magnetic order in dilute (~1%) alloys of the heavy rare-earth elements in Y. Such magnetism, occurring in alloys at concentrations far below the levels of nearest neighbor percolation, has confirmed the very long-range character of the indirect exchange interaction in the rare earths, and has conclusively demonstrated that a spin-glass state is not formed as suggested from previous indirect measurements.

■ In a cooperative project with the Smithsonian Institution, division scientists are using neutron autoradiography to study the painting techniques of master artists. Because neutron radiography can directly observe pigment applications beneath the surface of an oil painting, the researchers can retrace steps in the artist's creative process, from beginning line drawing, for example, to final brush stroke. The technique also provides information on the composition and origin of pigments, and can be used to judge the authenticity of artworks. Related work in the area of neutron NDE includes texture and residual stress studies of metals and ceramic composites.

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Theory and Instrumentation

The NIST research reactor features 25 state-of-the-art instruments for neutron-scattering materials analysis and neutron dosimetry studies. Because this equipment is not available in private-sector laboratories, a major goal of the division is to foster use of the instruments by outside researchers. RRD scientists serve as technical consultants, and they work to increase the utility of the research resource—by strengthening the theoretical underpinnings of neutron-scattering

methods and by developing new or improved instruments for experiments. Recent accomplishments include:

■ A significant advance in the theory of small-angle scattering which permits mathematical treatment of scattering from bicontinuous interfaces in microdispersed and microporous media. The RRD-developed simulation methods provide the first well-defined topological description of scattering from physical systems characterized by random morphologies with definite length scales, such as microemulsions and controlled-pore glasses.

■ Building on the theory of maximum entropy, RRD scientists have developed a mathematical approach that should be instrumental in determining the structures of biological macromolecules. The procedure extends the phases of diffraction data, creating high-resolution data from a low-resolution starting set.

■ Recent research to exploit neutron reflection and grazing angle diffraction as powerful probes of surface and interfacial structures has produced very promising results. As an example, a successful first study (with IBM) of lamellar formation and surface ordering in diblock copolymer films has been completed, and the ability to measure reflectivities as low as 10^{-6} demonstrated.

■ A new polarized-beam instrument has been developed jointly by NIST and the University of Maryland. It is used for determining magnetic states, based on changes in the phase of a polarized neutron beam.

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Databases

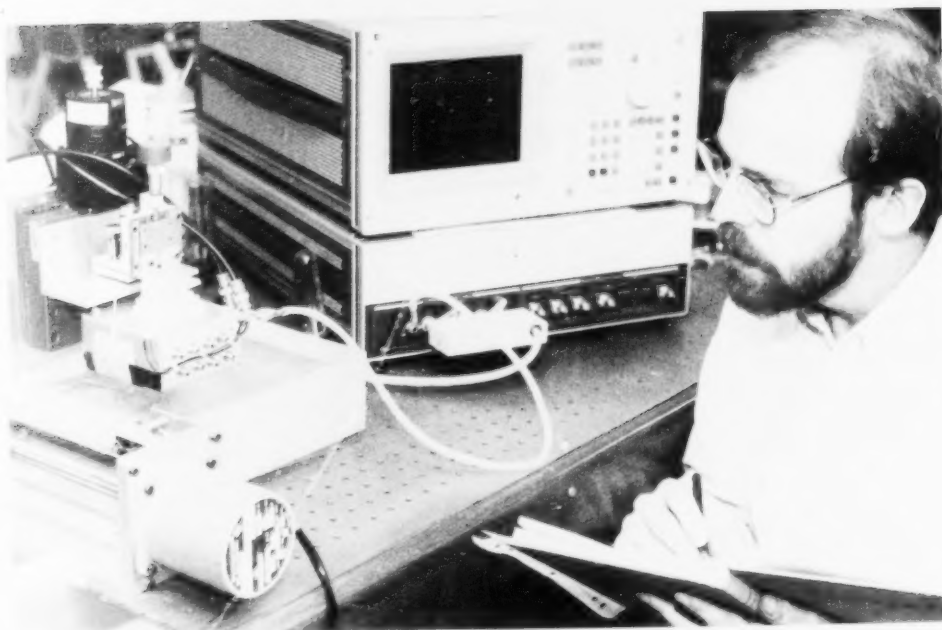
Located in the division, the NIST Crystal Data Center collects and critically evaluates data for two scientific databases: NIST Crystal Data and the NIST/Sandia/ICDD Electron Diffraction Database. These databases with over 140,000 entries contain chemical, physical, and crystallographic information on all classes of solid-state materials. They are made available to the scientific community via on-line search systems, CD-ROM discs, and on magnetic tape. A brief description of the on-line system and the adaptation of the Electron Diffraction Database to scientific instruments is given below.

■ On-line Search System: The Crystal Data Center and Canada's Scientific Numeric Database Service have officially combined efforts to make crystallographic data internationally available via CRYSDAT. Recently, new search and research tools have been built into the system. Usage has expanded, and more than 125 companies and research institutions use the system to solve analytical and research problems. For example, CRYSDAT has been extensively used in superconductor research to find materials with appropriate lattice parameters, space group, and chemical composition.

■ Electron Diffraction Database: This new database has been specifically designed for electron diffractionists. Currently, the database is being built into the workstations associated with several analytical electron microscopes. Unknown phases are identified by chemical/lattice matching procedures as soon as experimental elemental and d-spacing data have been recorded.

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Office of Nondestructive Evaluation



A newly developed capacitive array sensor is used to characterize minute subsurface defects in nonconductive materials.

Until recently, nondestructive evaluation (NDE) was principally concerned with in-service or post-manufacturing assessments of the integrity of structures or products. Such inspections—of nuclear reactors, aircraft, and pipelines, for example—are essential components of safety programs. Today, however, NDE methods are also employed as an essential ingredient of many manufacturing operations, monitoring important properties and characteristics of materials while they are being processed. In this capacity, NDE provides the information necessary for guiding or controlling production processes, assuring uniform high-quality products, and reducing waste.

The Office of Nondestructive Evaluation (ONDE) manages an agency-wide program for upgrading the quality of engineering materials and confirming their reliability in service. ONDE research activities capitalize on the broad range of talents and the wide variety of equipment available within MSEL and elsewhere in NIST.

In addition to its traditional role of developing NDE standards and methods, the office assembles interdisciplinary collaborations aimed at developing sensors and devices for nondestructive measurements. It funds and coordinates research on nondestructive evaluation in support of three key areas of advanced-materials processing: production and consolidation of ceramic and metal powders, forming of metals, and composites and interfaces. In these areas, the major aim is to develop NDE sensors and methods that provide the on-line measurements that can enable automated or intelligent processing. Examples of accomplishments in NDE research are included in the descriptions of some of the MSEL divisions. Others follow.

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Ceramic and Metal Powders

Studies on the production and consolidation of ceramic and metal powders are aimed at developing automated processing technologies for these materials. Within the NDE framework, sensors provide important information which is needed to develop and utilize expert systems for process control. They enable measurements of properties that

relate to the quality of the manufactured product, providing the basis for minimizing deviations during production.

■ The ongoing project on the formation and consolidation of metal powders (see page 4) demonstrates the evolution from the development of sensors to process control. The project, involving a consortium of researchers from NIST and private industry, began with the development and investigation of a variety of sensors. Selected sensors were installed in NIST's inert gas/metal atomization facility. Atomization experiments carried out with these sensors are guiding development of a process model and an expert system for intelligent control of the process.

■ Sponsored by ONDE, an NIST research team is investigating the potential of capacitive probes, ultrasonics, and AC spectroscopy for monitoring sintering of ceramics. This effort will likely be expanded to include eddy-current sensors, and a new class of ceramics—high T_c superconductors—will be investigated.

■ Thermal wave studies on cutting tools made of hot pressed silicon nitride were conducted to investigate machining damage in these specimens. These samples were subjected to grinding with various abrasion levels and measured after each grinding period. The average value of the thermal wave signals, when plotted as a function of signal penetration depth, showed definite correlation with the surface damage resulting from the grinding. Although these results are essentially qualitative, they suggest that more quantitative thermal wave measurements may provide a practical, nondestructive method for evaluating damage during machining of ceramics.

Forming of Metals

The goal of this activity in the NDE Program is to develop generic approaches, sensors, and procedures for characterizing metals

during the forming process. The emphasis is on measurements that can be made on the production line to improve process control rather than inspection techniques for post-manufacturing inspection.

Current efforts in this activity include the development of temperature sensors based on eddy-current techniques for determining the internal temperature distribution in hot metal objects (see page 5), an in-process ultrasonic monitor for grain texture in steel products, and ultrasonic sensors for on-line monitoring of both surface roughness and cutting-tool wear during machining.

■ The formability of sheet metal, which depends in large part on metallurgical texture or grain orientation, is a significant process variable in the manufacture of such diverse items as beverage cans, appliances, and automotive panels. In order to improve process control, a noncontacting ultrasonic method was developed last year for on-line monitoring of texture in aluminum alloy sheet. This year's research, in collaboration with the Advanced Steel Processing and Products Research Center, demonstrated that the technique is also applicable to thin ferritic steel sheet.

■ A means for determining surface roughness during machining of bulk metal parts is desirable for quality control of the finished product. An ultrasonic NDE sensor was developed 2 years ago for on-line monitoring of surface finish, and last year's research successfully demonstrated that the same ultrasonic sensor could be utilized on-line to detect worn or damaged cutting tools. This year's research showed that a finely focused ultrasonic beam can be used to map the topography of a surface in a fashion analogous to the contacting stylus profilometry technique.

Interfaces and Composites Processing

Because understanding of the characteristics of composites is far from

complete, development of NDE monitoring methods for these materials must often be preceded by studies that relate microstructural features (and the resultant properties) to the performance of the materials. This approach—collecting fundamental information on material properties and then proceeding to methods for detection, measurement, and, ultimately, process control—is demonstrated in the NDE work in the Polymers Division on monitoring of polymer melts. (See page 13.)

■ The characterization of interfaces is of vital importance in designing and manufacturing advanced composites. Theoretical and experimental research over the last 2 years has shown that ultrasonic waves propagating along the reinforcing fibers of a composite material radiate a fraction of their energy into the surrounding matrix, in the form of leaky ultrasonic waves. Research this year experimentally confirmed the quantitative aspects of a model for iron/aluminum and silicon carbide/aluminum metal-matrix composites.

Standards and Methods

When the Institute began the NDE program in the mid-1970's, the initial expectations were that standardization of NDE measurements could be completed in a few years. Instead, the need for NDE measurements traceable to national standards has grown tremendously. New NDE techniques are introduced almost continuously, and industry's growing interest in nondestructive measurements has spawned additional needs for



This laboratory set-up ultrasonically monitors the surface condition on a rotating part by means of sound echoed from the part surface.

standards and methods. For example, acoustic emission, capacitive NDE, real-time radiology, and thermography were either unknown or in their infancy 10 years ago. Today, industry and researchers regularly employ these methods and, in turn, the methods require standardization.

ONDE's long-term contract from the Army's Materials Technology Laboratory to develop new and improved military standards for NDE was especially fruitful this year. A standard field-worthy method for verifying the performance of acoustic emission (AE) testing systems is described in MIL-HDBK-786, prepared by researchers in the Center for Manufacturing Engineering (CME). A key feature of the method is the simulated AE event developed there previously and which earned an IR-100 award. Standard glossaries of terms and definitions for ultrasonic testing procedures and for radiographic testing were prepared in

CME and the Center for Radiation Research and were issued as Military Standards 371 and 369 respectively.

The American Society for Testing and Materials has initiated work on the development of a new standard test method to assess the visual acuity of inspectors and others who read and interpret radiographs. The basis of the standard is a set of 72 reference radiographs of artifacts containing tight discontinuities (artificial cracks) in different locations, orientations, contrasts, etc. The set was developed by scientists in the Center for Radiation Research, following a scheme conceived earlier in the Center for Building Technology. Although intended originally for radiographic applications, it is now anticipated that the new test method will also prove useful to practitioners of other NDE disciplines, such as visual inspection and liquid penetrant testing, where visual acuity is a critical factor.

As part of a project to prepare a standard method for qualifying eddy-current probes, a "standard crack" has been developed that can help the airline industry do a better job of gauging metal fatigue in the nation's aircraft fleet. NIST's standard crack is a 3-inch by 2-inch metal sample containing a well-characterized flaw that accurately simulates fatigue cracks. With it, researchers can determine if the probes used to detect fatigue cracks are working properly. The standard crack will be offered for sale through NIST's Standard Reference Materials program.